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(पहला पुनरीक्षण)

Indian Standard
SULPHITED FATLIQUORS FOR
LEATHER — SPECIFICATION
(*First Revision*)

ICS 59.140.10

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BUREAU OF INDIAN STANDARDS
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FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Leather, Tanning Materials and Allied Products Sectional Committee had been approved by the Chemical Division Council.

Sulphited fatliquors, also known as sulphited oil fatliquors are increasingly used in the leather processing on account of their remarkable stability of the water emulsion to various salts and acids used in leather processing. Sulphited oil fatliquors are produced by oxidative sulphitation of the oils by passing air through a mixture of oil and sodium bisulphite solution at the desired temperature. The important feature of the increased stability makes it possible to be used alongwith tanning materials during tanning.

This standard was originally published in 1998. In this revision, the requirement on pentachlorophenol (PCP) has been introduced keeping in view of the demand for eco-friendly inputs from the leather industry.

The composition of the Committee responsible for the formulation of this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960. 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SULPHITED FATLIQUORS FOR LEATHER — SPECIFICATION

(*First Revision*)

1 SCOPE

1.1 This standard prescribes requirements, methods of sampling and test for sulphited fatliquors used in the leather industry for fatliquoring.

1.2 This standard covers only sulphited fatliquors made from sulphited oils of marine and vegetable origin inclusive of any additives or blends of fatliquors made thereof.

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

<i>IS No.</i>	<i>Title</i>
548 (Part 1) : 1964	Methods of sampling and test for oils and facts: Part 1 Methods of sampling, physical and chemical tests
1070 : 1992	Reagent grade water (<i>third revision</i>)
1640 : 2007	Glossary of terms relating to hides, skins and leather (<i>first revision</i>)
4221 : 1967	Quality tolerances for water for tanning industry
14575 : 1999	Determination of pentachlorophenol (PCP) in leather — Method of test

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 548 (Part 1) and IS 1640 shall apply.

4 REQUIREMENTS

4.1 Physical Condition

The material shall be pourable at $27 \pm 2^\circ\text{C}$. Appearance of any graininess or a slight physical separation like a liquid and semi-solid portion appearing specially in winter season or long storage shall not be considered as defect so long as the two portions individually and together form ready emulsions in water.

4.2 Emulsion Characteristics

- a) The fatliquor shall form a stable emulsion in hot water (55°C - 65°C) when diluted in 1:10 ratio;
- b) The water emulsion containing 3 to 4 percent total fatty matter shall remain stable for at least 40 min at room temperature $27 \pm 2^\circ\text{C}$ without creaming and oil separation;
- c) The sulphited fatliquor shall be capable of being readily washed out with water without having any oily feeling in hand; and
- d) Ten percent emulsion shall remain stable for 40 min when mixed with 5 percent solutions of sodium chloride, calcium chloride, magnesium sulphate and 5 percent basic chromium sulphate solution in separate containers without creaming and oil separation.

4.3 Odour

The fatliquor shall be free from rancid or putrefactive odour of the oils.

4.4 Chemical Requirements

Sulphited oil fatliquors, besides meeting the characteristics mentioned under **4.1** to **4.3**, shall also meet the chemical requirements given in Table 1.

4.5 Keeping Quality

The fatliquor shall be stable for at least six months from its date of manufacture, when stored in original packing condition.

5 PACKING AND MARKING

5.1 Packing

The material after manufacture shall be packed in air-tight containers which shall not develop any rust or deterioration of the product due to the presence of water in the medium or any of the ingredients used in the formulation after storage of six months from the date of manufacture.

5.2 Marking

The containers shall be marked with the name of the

Table 1 Chemical Requirements
(Clause 4.4)

Sl No. (1)	Characteristic (2)	Requirement (3)	Method of Test, Ref to (4)
i)	pH of emulsion (1:10 dilution in distilled water)	6.5 - 7.5	A-2
ii)	Total active ingredient, percent by mass, <i>Min</i>	60	A-3
iii)	Unsaponifiable matter percent by mass, <i>Max</i>	15	A-4
iv)	Total alkalinity equivalent per 10 g, <i>Max</i> , in mg	5	A-5
v)	Total sulphur as sulphides (SO ₃), percent by mass, <i>Min</i>	1.8	A-6
vi)	Total ash, percent by mass, <i>Max</i>	4-5	A-7 as Referee method and A-8 as Routine method
vii)	PCP content ¹⁾ , mg/kg, <i>Max</i>	5	A-9

¹⁾ Calculated on moisture free basis.

manufacturer, trade-mark, if any; batch number; date of manufacture; and the quantity in kg.

5.2.1 BIS Certification Marking

The containers may also be marked with the Standard Mark.

5.2.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

ANNEX A

(Clause 4.4 and Table 1)

METHODS OF TEST

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, reagent grade chemicals and reagent grade water (*see* IS 1070) shall be used in tests.

A-1.2 Water to be used for tests other than chemical tests (*see* 4.4 and Table 1) shall conform to IS 4221.

A-2 DETERMINATION OF pH OF WATER EMULSION

A-2.1 Apparatus — pH meter, electrode type.

A-2.2 Procedure

Prepare 10 percent emulsion by dispersing 10 g of fatliquor in 90 ml water. Determine the pH by using a suitable pH meter.

NOTE — As the pH of solution of sulphited oil fatliquors varies with the temperature, it should be controlled during the tests, and in some cases it may be necessary to carry out the test at 50°C.

A-3 DETERMINATION OF TOTAL ACTIVE INGREDIENT

A-3.1 Reagents

A-3.1.1 Ethyl Alcohol

A-3.1.2 Petroleum Ether (Boiling Range 40°C-60°C)

A-3.2 Procedure

Weigh about 5 g of the sulphited oil fatliquor accurately in a 250 ml flask and add 25 ml of 50 percent ethyl alcohol and 25 ml of petroleum ether. Transfer the contents to a separating funnel. Shake the contents vigorously and allow the layers to separate. Transfer the lower alcohol layer to another separating funnel and extract it three or four times with petroleum ether. Extract upper layer, namely, the petroleum ether layer, with 75 percent alcohol and allow the layers to separate and extract further with 90 percent alcohol and absolute alcohol and allow the layers to separate.

Collect the petroleum ether layer and alcohol layer in two tared flasks. Evaporate the solvents, dry the residues to constant weight in the oven, cool and weigh.

Alcohol layer contains the emulsifier and the petroleum ether layer contains the neutral oil.

$$\text{Free oil, in percent (A)} = \frac{F}{W} \times 100$$

$$\text{Emulsifier in percent (B)} = \frac{E}{W} \times 100$$

where

F = weight of petroleum ether solubles, in g;

E = weight of alcohol solubles, in g; and

W = weight of the fatliquor sample, in g.

Total active ingredient in percent = $A + B$

A-4 DETERMINATION OF UNSAPONIFIABLE MATTER

The unsaponifiable matter of sulphited fatliquors shall be determined in accordance with the procedure given in 8 of IS 548 (Part 1).

A-5 DETERMINATION OF TOTAL ALKALINITY

A-5.1 Procedure

Dissolve 10 g of the sample in 100 ml of water in a conical flask, warm, if necessary, to obtain solution. After cooling, add 30 g of sodium chloride, 25 ml of ether and 5 drops of 0.1 percent methyl orange indicator and titrate with 0.5 N sulphuric acid until the aqueous layer is orange. Stopper the flask and shake the contents frequently during the titration.

A-5.2 Calculation

Calculate the total alkalinity as follows:

$$A = \frac{10 \times V \times N}{W}$$

where

A = total alkalinity in mg equivalent per 10 g of oil;

V = volume of sulphuric acid, in ml;

N = normality of sulphuric acid; and

W = weight of the sample, in g;

NOTE — This method is not applicable in case sodium acetate or other compounds which are not accurately titratable to methyl orange in aqueous solution is present in the fatliquor.

A-6 DETERMINATION OF TOTAL SULPHUR AS SULPHITE (SO_3)

A-6.1 Apparatus

A-6.1.1 Muffle Furnace

A-6.1.2 Platinum Dish

A-6.2 Reagents

A-6.2.1 Sodium Carbonate

A-6.2.2 Potassium Chlorate

A-6.2.3 Hydrochloric Acid, Dilute and Concentrated

A-6.2.4 Barium Chloride, 5 percent aqueous solution (w/w).

A-6.2.5 Methyl Orange Indicator

A-6.3 Procedure

Weigh accurately about 2 g of the sulphited oil fatliquor in a platinum dish. Add 5 g of sodium carbonate and 1 g of potassium chlorate and 5 ml of distilled water to it and heat the same using a low flame. Ash the mass in a muffle furnace at 700°C - 750°C . Transfer the sulphated ash to a beaker washing with dilute hydrochloric acid. Add ammonia till the solution turns slightly alkaline. Add methyl orange indicator and make it up to 200 ml. Take 50 ml of the solution in a beaker, add 1 ml of concentrated hydrochloric acid and 10 percent barium sulphate. Filter the precipitate in a low ash filter paper, dry and ash in a tared crucible. Dry and ash in the air oven, cool and weigh.

A-6.4 Calculation

$$\text{Total sulphur as sulphite, in percent} = \frac{w \times 80}{233.7 \times W} \times 100$$

where

w = weight of barium sulphate, in g; and

W = weight of the fatliquor sample, in g.

A-7 DETERMINATION OF TOTAL ASH (REFEREE METHOD)

A-7.1 Apparatus

A-7.1.1 Muffle Furnace

A-7.1.2 Platinum Crucible

A-7.2 Procedure

Weigh 3 g of sample in a platinum crucible and heat gently until a charred residue remains. Extract this with hot water to remove soluble salts, filter through a low ash filter paper and wash thoroughly. Ash the filter

paper and charred residue, moistening with ash-free hydrogen peroxide, if any difficulty is experienced. Allow the dish to cool. Add the aqueous extract and evaporate on a steam bath. Finally complete the ashing at as low a temperature as possible. Cool the dish and weigh.

A-7.3 Calculation

Calculate the ash content in the following manner:

$$\text{Ash content in percent} = \frac{W_2 - W_0}{W_1 - W_0} \times 100$$

where

W_0 = weight of empty, dry crucible, in g;

W_1 = weight of crucible containing the sample before ashing, in g; and

W_2 = weight of the crucible containing the residue after ashing, in g.

A-8 DETERMINATION OF TOTAL ASH (ROUTINE METHOD)

A-8.1 Apparatus

A-8.1.1 Muffle Furnace

A-8.1.2 Silica Crucible

A-8.2 Procedure

Weigh about 2 g of the sample accurately in a dry and clean silica crucible. Heat the crucible on a low, oxidizing flame till all the carbon containing material in a beaker, add 1 ml of concentrated hydrochloric acid is oxidized. Place the crucible in a muffle furnace and 10 percent barium chloride solution till the whole maintained at a temperature of 800°C for about 3 h. Remove the crucible from the muffle furnace and place it on an asbestos sheet for about 5 min and then transfer the same into a closed desiccator. Weigh the crucible containing the ash after it attains the room temperature.

A-8.3 Calculation

Calculate the ash content in the following manner:

$$\text{Ash content in percent} = \frac{W_2 - W_0}{W_1 - W_0}$$

where

W_0 = weight of empty, dry crucible, in g;

W_1 = weight of crucible containing the sample before ashing, in g; and

W_2 = weight of the crucible containing the residue after ashing, in g.

NOTE — For the purpose of routine analysis the method prescribed in A-8 shall be used. However in case of any dispute the method prescribed in A-7 shall be used as the referee method.

A 9 DETERMINATION OF PENTACHLORO-PHENOL (PCP)

A-9.1 Transfer about 5 g of the sulphated oil into a separating funnel of about 250 ml capacity along with 100 ml water. Add 15-25 ml of sulphuric acid 10 percent (w/v) aqueous solution and ensure that the pH of the contents is acidic. Then carry out liquid-liquid extraction using hexane, analytical grade, as the solvent. At first extract with about 25 ml hexane by shaking the contents thoroughly for about 15 min. Then set it aside for clear separation of the layers (*see* Note). Transfer the aqueous layer into another separating funnel and carryout three more extractions of the aqueous layer with hexane of 25 ml each time in the same manner explained earlier.

NOTE — In case of any emulsion being noticed, break the same with the addition of 20-30 ml saturated sodium chloride aqueous solution or follow the steps explained in 4.3.4.2 of IS 14575.

A-9.2 Combine the hexane extracts and dry it by passing through a bed of anhydrous sodium sulphate of about 10 g. The dried hexane layer is evaporated to near dryness by a rotary evaporator.

A-9.3 The residue is taken for acetylation. Then follow the procedure as detailed in Method A of IS 14575.

ANNEX B*(Foreword)***COMMITTEE COMPOSITION**

Leather, Tanning Materials and Allied Products Sectional Committee, CHD 17

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Ministry of Commerce, New Delhi	REPRESENTATIVE

IS 14488 : 2013

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